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54 **Peroxyacid bleach composition and its use in detergent compositions.**

57 A stable, dry, granular peroxyacid bleach composition having particle size of from 500-2000 μm is disclosed, said composition comprising from 20 to 65% by weight of a solid organic peroxyacid compound, 79.5 to 30% by weight of at least one inert inorganic salt, and from 0.5 to about 6.5% by weight of a non-oxidisable, acidic, polymeric builder.

Low to medium temperature bleach detergent compositions comprising a surface-active agent, a detergency builder and said dry granular peroxyacid bleach composition are also disclosed.

EP 0 256 443 A2

PEROXYACID BLEACH COMPOSITION AND ITS USE IN DETERGENT BLEACH COMPOSITIONS

This invention relates to a dry, granular bleaching composition comprising a peroxyacid compound, having improved stability and effectiveness. The dry, granular peroxyacid bleaching composition of the invention can be used as a bleach additive to or as a bleach component in detergent compositions suitable for washing, cleaning, sanitizing and/or bleaching of substrates, particularly fabrics.

5 Organic peroxyacids as a class are quite effective bleaches and the use of organic peroxyacid compounds as the bleach system in detergent compositions has been proposed in the art (see for example GB Patent 1,456,591, US Patent 4,100,095 and EP-B-00 37 146). Although the efficiency of certain organic peroxyacid compound bleach systems has long been recognized at low to medium wash temperatures, the major problem, which has prevented their exploitation so far, has been their inherent instability in
10 conventional alkaline fabric-washing compositions. Their use has therefore been restricted to specialized wash additive products.

Various attempts have been made to resolve the instability problem, the basic concept being mixing, granulating and/or coating the organic peroxyacid particles with a surfactant compound, an exotherm control agent or an acid diluent, as described in US Patent 4,126,573 and 4,100,095; or by providing a mixture of
15 the organic peroxyacid with a buffering agent to control the pH of the aqueous solution, as described in US Patents 4,170,453 and 4,259,201; or by using a stabilizing system comprising e.g. magnesium and alkali metal sulphates, such as described in GB Patent 1,456,591.

None of the protection routes as outlined above appeared to be satisfactory, and no-one has yet been able to successfully incorporate a reactive peroxyacid in alkaline fabric-washing powder compositions.

20 However, the efficiency of direct peroxyacid bleaching systems, i.e. not formed *in situ* from a precursor and a percompound, at low to medium wash temperatures, combined with their insensitivity to catalase and peroxidase systems, has made them extremely attractive options for use in the next generation of fabric-washing products.

Solid peroxyacid, particularly diperoxy dodecanedioic acid, in highly diluted forms of granules, powder
25 mix or aqueous suspensions having a peroxyacid content of up to 12% by weight have been manufactured on a small scale. Such highly diluted organic peroxyacid forms are, however, not suitable for use as an effective bleach system in detergent bleach compositions owing to high amounts of inactive material introduced into the composition.

The present invention seeks to provide an improved granular organic peroxyacid bleach composition
30 which can be suitably used as an effective bleach component of fabric-washing compositions.

It has now surprisingly been found that it is possible to obtain an effective dry, granular peroxyacid bleach composition comprising from 20 to 65% by weight of organic peroxyacid, which is remarkably stable for use in alkaline fabric-washing compositions.

The dry, granular peroxyacid bleach composition of the invention consists of particles of which more
35 than 75% by weight will have a size of from 500-2000µm, and comprises from 20 to 65% by weight of an organic peroxyacid compound, at least one inert inorganic salt in an amount of from 79.5-30% by weight, and from 0.5 to about 6.5% by weight of a non-oxidisable acidic polymeric binder.

Selection of the correct polymer binder is of crucial importance and must be controlled by its resistance to oxidation and its acid pH, i.e. below 7. The acidic polymers usable as a binder in this invention can be
40 any non-cellulosic homo-or copolymeric mono-and polycarboxylic acids having an average molecular weight of from 500 to about 1,000,000, preferably from 2000-250,0000, more preferably from 10,000-50,000.

Suitable polymers include those derived from acrylic acid, methacrylic acid, maleic acid, citraconic acid, aconitic acid, fumaric acid, mesaconic acid, phenyl maleic acid, benzyl maleic acid, itaconic acid, methylene malonic acid, alpha-C₁-C₄ alkyl acrylic acid, alpha-hydroxy acrylic acid and acetalcarboxylic acid
45 monomers, or from the anhydrides of the above monomers where these exist. The polymer can be homopolymers of the above mono-or polycarboxylic monomers; or copolymers of two or more of the above mono-or polycarboxyl monomers; or copolymers of one or more of the above carboxyl monomers with an unsaturated polymerizable monomer other than the specified mono-and polycarboxyl monomers; or modified homo-or copolymers of the above classes having for example a non-oxidisable phosphinic acid or
50 sulphinic acid group.

Preferred acidic polymeric binders are polyacrylic acids, phosphinate-modified polyacrylic acids such as described in GB Patents 1,485,235 and 1,595,688 and EP-A-01 82 411; copolymers of maleic acid (anhydride) and acrylic or methacrylic acid; and acidic copolymers containing hydrophobic groups, such as copolymers based on polymethacrylic acid and polyacrylic acid esters in which the ratio of free carboxyl groups to ester groups is at least 1:1.

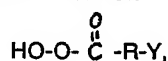
Copolymers of the latter group can be advantageously used as polymeric binder for granular peroxyacid bleach compositions for use in liquid bleaching compositions, where they could further reduce the solubility of the organic peroxyacid, and hence improve its stability.

Although changes in molecular weight did not appear to markedly influence the peroxyacid stability, they are of some influence on the granule's physical properties, e.g. particle strength, resistance to attrition and film-forming properties. Preferred molecular weights are from 2000-250,000, more preferably from 10,000-50,000.

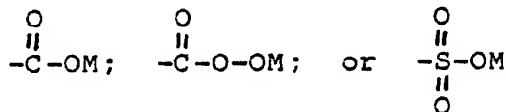
The inert inorganic salts usable in the present invention are non-alkaline salts and can be neutral or acid salts. They are used as a diluent in an amount within the range as specified above, which is adjustable at will dependent upon the levels of the organic peroxyacid compound and the acidic polymeric binder in the granule composition. Examples of such suitable inert inorganic salts are sodium sulphate (Na_2SO_4), sodium hydrogen sulphate (NaHSO_4), potassium sulphate (K_2SO_4), potassium hydrogen sulphate (KHSO_4); the alkali metal acid phosphates such as Na_2HPO_4 , NaH_2PO_4 , K_2HPO_4 and KH_2PO_4 .

The organic peroxyacid compounds used in the present invention are solid at room temperature and should preferably have a melting point of at least 50°C .

Such peroxyacid compounds are the organic peroxyacids and water-soluble salts thereof having the general formula:

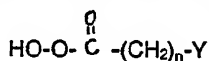


wherein R is an alkylene or substituted alkylene group containing 1 to 20 carbon atoms or an arylene group containing from 6 to 8 carbon atoms, and Y is hydrogen, halogen, alkyl, aryl or any group which provides an anionic moiety in aqueous solution. Such Y groups can include, for example:

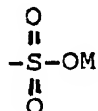


wherein M is H or a water-soluble, salt-forming cation.

The organic peroxyacids and salts thereof usable in the present invention can contain either one, two or more peroxy groups and can be either aliphatic or aromatic. When the organic peroxyacid is aliphatic, the unsubstituted acid may have the general formula:

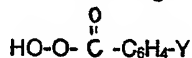


wherein Y can be H, $-\text{CH}_3$, $-\text{CH}_2\text{Cl}$, $-\overset{\text{O}}{\overset{\parallel}{\text{C}}}-\text{OM}$,

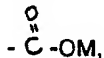


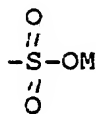
or $-\overset{\text{O}}{\overset{\parallel}{\text{C}}}-\text{O}-\text{OM}$ and n can be an integer from 6 to 20. Peroxydodecanoic acids, peroxytetradecanoic acids and peroxyhexadecanoic acids are the most preferred compounds of this type, particularly 1, 12-diperoxydodecanedioic acid, 1,14-diperoxytetradecanedioic acid and 1,16-diperoxyhexadecanedioic acid. Examples of other preferred compounds of this type are diperoxyazelaic acid, diperoxyadipic acid and diperoxysebacic acid.

When the organic peroxyacid is aromatic, the unsubstituted acid may have the general formula:



wherein Y is, for example, hydrogen, halogen, alkyl,





5 or - $\overset{\text{O}}{\parallel} \text{C} - \text{O} - \text{OM}$.

The percarboxy and Y groupings can be in any relative position around the aromatic ring. The ring and/or Y group (if alkyl) can contain any non-interfering substituents such as halogen or sulphonate groups. Examples of suitable aromatic peroxyacids and salts thereof include monoperoxyphthalic acid, diperoxyterephthalic acid, 4-chlorodiperoxyphthalic acid, diperoxyisophthalic acid, peroxy benzoic acids and ring-substituted peroxy benzoic acids, such as peroxy- α -naphthoic acid. A preferred aromatic peroxyacid is diperoxyisophthalic acid.

The granular peroxyacid bleach composition of the invention will preferably have a particle size of between 500 and 1700 μm , more preferably from 500 μm to 1200 μm , and a reactive organic peroxyacid content of from 35% to about 55% by weight. The level of acidic polymeric binder should preferably be kept as low as possible, e.g. from 0.5-3.0% by weight, as true encapsulation is not aimed at, in contrast with previous workers who believed in the theory of stabilization by encapsulation. Besides, high binder levels were found to reduce peroxyacid stability.

though not essential, a heavy metal sequestrant, such as ethylenediamine tetraacetic acid (EDTA), ethylenediamine tetramethylene phosphonic acid (EDTMP), dipicolinic acid or Chel® DP may further be incorporated as desired in the peroxyacid granule of the invention. They are normally incorporated in only small amounts of up to about 5% by weight of the composition, preferably from 0.05 to 2.5% by weight.

The granular peroxyacid bleach composition of the invention may further contain any solid organic acid, such as citric acid, succinic acid, adipic acid, glutaric acid and mixtures thereof. These may be present at a level of up to about 20% by weight of the composition.

Additional coating of the peroxyacid granule with a protective layer does not essentially contribute to increased peroxyacid stability, but it may be applied as desired to prevent or reduce interaction with oxidation-susceptible components of the detergent composition, such as fluorescers and perfumes.

Suitable coating materials are, for example, saturated long-chain fatty acids of C_{12} - C_{22} chain length. These coating materials can be advantageously used at a level of 1-5% by weight. In most cases a coating level of about 2% by weight is adequate for serving the purpose and there is no need to exceed said level.

The dry, granular peroxyacid bleach composition can be conveniently prepared by spraying polymeric binder solutions onto a dry mixture of the organic peroxyacid and the inert inorganic salt using any type of granulating equipment, such as a pan granulator or a rotating drum-type granulator. Alternatively, the other granulation processes, such as noodling, marumerizing and the like can be used, followed by drying.

If any of the above-mentioned other ingredients are present, these can be included in the dry mixture of peroxyacid and inorganic salt.

Optional coating can be applied by a second spray of liquefied coating material onto the dry, granular composition.

As explained before, the dry, granular peroxyacid bleaching composition of the invention can be used as an effective bleach component in detergent compositions, which are particularly suitable for use at low to medium wash temperatures, e.g. from 20°C to 60°C.

Accordingly, in another aspect of the invention a low to medium temperature bleach detergent composition is provided comprising a surface-active compound, a detergency builder and a dry granular peroxyacid bleaching composition as defined hereinbefore.

Said dry, granular peroxyacid bleaching composition of the invention can be incorporated in bleach detergent compositions in amounts of from 1 to 15% by weight, preferably from 2 to 10% by weight, calculated as organic peroxyacid, without difficulty.

The bleach detergent compositions of the invention will contain at least one surface-active compound, which may be anionic, cationic, nonionic or amphoteric in character, the amount of which generally will be from about 3 to about 40%, preferably from 10 to 35% by weight.

Generally, mixtures of the above surface-active compounds are used; mixtures of anionic and nonionic surface-active compounds are commonly used.

Typical anionic surface-active compounds are water-soluble or water-dispersible salts of various organic acids. The cations of such salts are generally alkali metals, such as sodium and, less preferably, potassium, but other cations, such as ammonium and substituted ammonium, can be used if desired. Examples of suitable organic acids are: alkyl benzene sulphonic acids, the alkyl chains of which contain from about 8 to

about 20 carbon atoms, such as p-dodecyl benzene sulphonic acid and linear alkyl (C₁₀₋₁₅) benzene sulphonic acid; the mixtures of sulphonic acids obtained by reacting linear and branched olefins, particularly linear "cracked-wax" or "Ziegler" alpha-olefins, containing from about 8 to about 22 carbon atoms, with sulphur trioxide; alkyl sulphonic acids obtained by reacting alkanes containing from about 8 to about 22 carbon atoms with sulphur dioxide/oxygen or sulphur dioxide/chlorine (followed by hydrolysis in the latter case), or by the addition of bisulphite to olefins, particularly linear "cracked-wax" or "Ziegler alpha-olefins, containing from about 8 to about 22 carbon atoms; alkyl sulphuric acids obtained by reacting aliphatic alcohols containing from about 8 to about 22 carbon atoms with sulphur trioxide; alkyl ether sulphuric acids, obtained by reacting molar quantities of aliphatic alcohols containing from about 6 to about 18 carbon atoms with from about 1 to about 15 moles of ethylene oxide, or a suitable mixture of ethylene oxide and propylene oxide, and subsequently reacting the alkoxyated alcohol with sulphur trioxide to yield the required acid; and natural or synthetic aliphatic carboxylic acids, particularly those derived from natural sources, such as tallows, coconut oil, palm oil, palm kernel oil and groundnut oil.

Examples of suitable nonionic surface-active compounds are condensates of alkylphenols having an alkyl group (derived, for example, from polymerized propylene, diisobutylene, octene, dodecene or nonene) containing from about 6 to 12 carbon atoms in either a straight-chain or branched-chain configuration, with about 5 to 25 moles of ethylene oxide per mole of alkylphenol; condensates containing from about 40% to about 80% polyoxyethylene by weight and having a molecular weight of from about 5,000 to about 11,000 resulting from the reaction of ethylene oxide with the reaction product of ethylenediamine and excess propylene oxide; condensates of linear or branched-chain aliphatic alcohols containing from 8 to 18 carbon atoms with ethylene oxide, e.g. a coconut alcohol-ethylene oxide condensate containing about 6 to 30 moles of ethylene oxide per mole of coconut alcohol; long-chain tertiary amine oxides corresponding to the general formula R₁R₂R₃N O, wherein R₁ is an alkyl radical containing from about 8 to 18 carbon atoms and R₂ and R₃ are each methyl, ethyl or hydroxy ethyl radicals, such as dimethyldodecylamine oxide, dimethyloctylamine oxide, dimethylhexadecylamine oxide and N-bis (hydroxyethyl) dodecylamine oxide; long-chain tertiary phosphine oxides corresponding to the general formula RR'R''P=O, wherein R is an alkyl, alkenyl or monohydroxyalkyl radical containing from 10 to 18 carbon atoms and R' and R'' are each alkyl or monohydroxyalkyl groups containing from one to three carbon atoms, such as dimethyldodecylphosphine oxide, dimethyloctadecylphosphine oxide, ethylmethyl-tetradecylphosphine oxide, dimethylstearylphosphine oxide, ethylpropylcetylphosphine oxide, diethyl-dodecylphosphine oxide, bis (hydroxymethyl) dodecylphosphine oxide, bis (2-hydroxyethyl) dodecylphosphine oxide, 2-hydroxypropylmethyl-tetradecylphosphine oxide, dimethylololeylphosphine oxide and dimethyl-2-hydroxydodecylphosphine oxide; and dialkyl sulfoxides corresponding to the general formula RR'S O, wherein R is an alkyl, alkenyl, beta-or gamma-monohydroxyalkyl radical or an alkyl or beta-or gamma-monohydroxyalkyl radical containing one or two other oxygen atoms in the chain, the R groups containing from 10 to 18 carbon atoms and wherein R' is a methyl, ethyl or alkylol radical, such as dodecyl methyl sulfoxide, tetradecyl methyl sulfoxide, 3-hydroxytridecyl methyl sulfoxide, 2-hydroxydodecyl methyl sulfoxide, 3-hydroxy-4-dodecyloxybutyl methyl sulfoxide, 2-hydroxy-3-decyloxypropyl methyl sulfoxide, dodecyl ethyl sulfoxide, 2-hydroxydodecyl ethyl sulfoxide and dodecyl-2-hydroxyethyl sulfoxide.

Examples of suitable amphoteric surface-active compounds are: derivatives of aliphatic secondary and tertiary amines, in which the aliphatic radical may be straight-chain or branched and wherein one of the aliphatic substituents contains from about 8 to 18 carbon atoms and one contains an anionic water-solubilizing group, such as sodium-3-dodecylaminopropionate, sodium-3-dodecylaminopropanesulphonate and sodium N-2-hydroxydodecyl-N-methylaurate; and derivatives of aliphatic quaternary ammonium compounds, sulphonium compounds and phosphonium compounds in which the aliphatic radical may be straight-chain or branched and wherein one of the aliphatic substituents contains from about 8 to 18 carbon atoms and one contains an anionic water-solubilizing group, such as 3-(N,N-dimethyl-N-hexadecylammonium) propane-1-sulphonate betaine, 3-(N,N-dimethyl-N-hexadecylammonium)-2-hydroxy-propane-1-sulphonate betaine, 3-(dodecylmethylsulphonium) propane-1-sulphonate betaine, and 3-(cetyl-methylphosphonium) ethane sulphonate betaine.

Further examples of suitable surface-active compounds commonly used in the art are given in "Surface Active Agents", Volume I, by Schwartz and Perry (Interscience 1949) and "Surface Active Agents", Volume II, by Schwartz, Perry and Berch (Interscience 1958).

The bleach detergent composition of the invention also includes one or more detergency builders. Usually the total amount of detergency builders in a detergent composition of the invention will be from about 5% to about 70% by weight of the detergent composition. Many detergency builders are known, and those skilled in the art of formulating fabric-washing detergent compositions will be familiar with these

materials. Examples of known detergency builders are sodium tripolyphosphate; sodium orthophosphate; sodium pyrophosphate; sodium trimetaphosphate; sodium ethane-1-hydroxy-1,1-diphosphonate; sodium carbonate; sodium silicate; sodium citrate; sodium oxydiacetate; sodium nitrilotriacetate; sodium ethylenediaminetetraacetate; sodium salts of long-chain dicarboxylic acids, for instance straight-chain (C₁₀ to C₂₀) succinic acids and malonic acids; sodium salts of alpha-sulphonated long-chain monocarboxylic acids; sodium salts of polycarboxylic acids; i.e. acids derived from the polymerization or copolymerization of unsaturated carboxylic acids and unsaturated carboxy acid anhydrides such as maleic acid, acrylic acid, itaconic acid, methacrylic acid, crotonic acid and aconitic acid, and the anhydrides of these acids, and also from the copolymerization of the above acids and anhydrides with minor amounts of other monomers, such as vinyl chloride, vinyl acetate, methyl methacrylate, methyl acrylate and styrene; and modified starches such as starches oxidized, for example using sodium hypochlorite, in which some anhydroglucose units have been opened to give dicarboxyl units.

Another type of detergency builder which can be used, either alone or in admixture with other builders, is a cation-exchange material, especially a sodium aluminosilicate such as described in GB Patent 1,429,143 or in Netherlands Patent Application 7403381.

Preferred materials of this type have the formula:



and may be amorphous or crystalline, with some bound water usually in an amount of about 10-30% depending on the drying conditions used. Such water-insoluble aluminosilicate cation-exchange materials are available as very finely divided powder, commercially known under various grades of zeolites, e.g. Zeolite A and Zeolite B.

Any of these builders can be used to prepare the detergent base powder for admixing with the dry, granular peroxyacid bleaching composition of the invention. Preferably the solution pH of the base powder measured at 5 g/l in distilled water should not exceed 9.5 and the moisture content is kept low.

This is of particular importance in the case of non-phosphorus detergent compositions comprising sodium carbonate as the main builder component. In such bleach detergent compositions the detergent base powder should preferably have a moisture content of not more than 6%, particularly not more than 3%.

If zeolite-built detergent compositions are used as the base powder, the dry, granular peroxyacid bleaching composition should preferably incorporate a heavy metal sequestrant. Furthermore, acidic copolymers containing hydrophobic groups are preferably used as the acidic polymeric binder for improved stability.

Optionally and preferably, the zeolite-built detergent base powder composition incorporates a hydratable material to act as moisture sink.

In addition to the above-described surface-active compounds and builders, the bleach detergent composition of the invention may contain any other non-interfering ingredients normally used in detergent compositions in minor amounts, which serve to improve the bleaching and laundering characteristics of the compositions or which add aesthetic appeal to the composition. Such minor ingredients can include buffering agents, sequestering agents and co-builders; suds-control agents; soil-suspending agents and anti-redeposition agents; enzymes, particularly proteolytic and lipolytic enzymes; corrosion inhibitors; optical brighteners; colouring agents, perfumes and filler materials. Generally, such minor components comprise no more than 20% by weight of the bleach detergent composition. Furthermore, peroxide bleaches such as sodium perborate may also be included with benefit in amounts from e.g. 2-20% by weight, preferably from 5-15% by weight of the detergent bleach composition.

Examples

The following dry, granular peroxyacid bleaching compositions were prepared on a laboratory rig (pan diameter = 14 cm). The pitch and speed of rotation could both be varied. Typical settings were a spindle angle of 55° to the horizontal and a speed of 80 rpm. Binder solutions were sprayed onto mixtures of diperoxydodecanedioic acid (DPDA) and sodium sulphate from a Humbrol® paint atomiser.

The granulated products were dried (over P₂O₅ or at 40°C) and sieved to obtain the 500-1700µm fractions.

	<u>Granular</u>	<u>DPDA</u>	<u>Binder</u>	<u>Molecular</u>	<u>Surface</u>
	<u>Bleach</u>	<u>Concen-</u>	<u>Concen-</u>	<u>Weight</u>	<u>Area</u>
5	<u>Composition</u>	<u>tration (%)</u>	<u>tration (%)</u>	<u>Binder</u>	<u>(m²/g)</u>
	I	49.0	PAA (0.95)	250,000	-
	II	49.0	PAA (1.83)	250,000	-
10	III	32.6	PAA (0.62)	250,000	0.9
	IV	49.0	DKW (1.3)	1,500	-
	V	32.6	DKW (0.95)	1,500	0.9

PAA = Polyacrylic acid

15 DKW = Phosphinate-modified polyacrylic acid.

Surface area was determined by nitrogen absorption and desorption onto and from a known weight of substrate (Quantasorb® apparatus).

Granule compositions III and IV were each dry-mixed with the following base powder composition (A) at a ratio of 82% base powder to 18% granule composition.

20	<u>Base Powder Composition (A)</u>	<u>% by weight</u>
	Sodium dodecyl benzene sulphonate	12.0
25	Nonionic ethylene oxide adduct	7.0
	Sodium soap	1.0
	Lather modifier	0.5
	Sodium triphosphate	32.0
30	Neutral silicate	4.3
	Sodium carboxymethylcellulose	1.6
	Hydroxyethylcellulose	0.3
35	Fluorescer	0.14
	Na ₂ SO ₄	32.76
	Water	8.4

40 The powders were stored in open vials at 30°C and 75% relative humidity conditions and the peroxyacid decomposition rates of both compositions were monitored.

The results showed peroxyacid losses of less than 25% after 60 days.

45 When the experiments were repeated with granule compositions I and II, i.e. both using PAA as binder at a level of 0.95% and 1.83% respectively, the same results were obtained, from which it can be concluded that the peroxyacid decomposition was not affected by changes in binder level over the test range of 0.6 to 1.8%

50 Claims

1. Dry, granular peroxyacid bleach composition having particle size of from 500-2000 µm and comprising from 20 to 65% by weight of a solid organic peroxyacid compound, 79.5 to 30% by weight of at least one inert inorganic salt, and from 0.5 to about 6.5% by weight of a non-oxidisable acidic polymeric binder.

55 2. Peroxyacid bleach composition according to claim 1, characterized in that the acidic polymeric binder is selected from the group of non-cellulosic homo-or copolymeric mono-and polycarboxylic acids having an average molecular weight of from 500 to about 1,000,000.

3. Peroxyacid bleach composition according to claim 2, characterized in that said acidic polymeric binder has an average molecular weight of from 2000 to 250,000.

4. Peroxyacid bleach composition according to claim 1, 2 or 3, characterized in that said acidic polymeric binder is polyacrylic acid or a phosphinate-modified polyacrylic acid.

5. Peroxyacid bleach composition according to any one of the above claims, characterized in that said inert inorganic salt is a non-alkaline neutral or acid salt.

6. Peroxyacid bleach composition according to any one of the above claims, characterized in that it comprises from 35 to 55% by weight of said peroxyacid compound.

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